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THE SHEAR AND THERMAL STRESS BEHAVIOR OF A SUPERREFINED MINERAL OIL AND A FLUOROSILICONE

FREDRICK C. BROOKS

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AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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Two high temperature hydraulic fluids were characterized as to their shear and thermal stress behavior in the environment of a new small-volume high-temperature evaluation stand. A dewaxed superrefined mineral oil (MIL-H-27601) was investigated at 550, 600, 625, and 700°F and exhibited no significant degradation to 600°F in the inerted system environment. A modified fluorosilicone (MLO-70-79) was investigated at 600°F and experienced a significant loss in viscosity (100°F) after 58 hours of environmental residence.

A detailed analysis was made of the results of these characterizations and the results of previous studies with the same fluids in a large volume system. From the analysis of these data it is shown that, for similar test profiles, the two circuits produce fluid property changes which correlate with the induced environments. Relationships between operational features in the two fluid systems and differences in fluid results are established.

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FOREWORD

This report was prepared by the Fluid and Lubricant Materials Branch, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. This work was conducted under Project Number 7340, "Nonmetallic and Composite Materials", Task Number 734008, "Energy Transfer Fluids", with F. C. Brooks acting as Project Engineer.

This report presents the purpose and results of an effort expended between October 1970 and April 1971.

This report was submitted by the author in June 1971.

This technical report has been reviewed and is approved.

R. L. Adamczak

R. L. ADAMCZAK, CHIEF
Fluid & Lubricant Materials Branch
Nonmetallic Materials Division

ABSTRACT

Two high temperature hydraulic fluids were characterized as to their shear and thermal stress behavior in the environment of a new small-volume high-temperature evaluation stand. A dewaxed superrefined mineral oil (MIL-H-27601) was investigated at 550, 600, 625, and 700°F and exhibited no significant degradation to 600°F in the inerted system environment. A modified fluorosilicone (MLO-70-79) was investigated at 600°F and experienced a significant loss in viscosity (100°F) after 58 hours of environmental residence.

A detailed analysis was made of the results of these characterizations and the results of previous studies with the same fluids in a large volume system. From the analysis of these data it is shown that, for similar test profiles, the two circuits produce fluid property changes which correlate with the induced environments. Relationships between operational features in the two fluid systems and differences in fluid results are established.

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SECTION I

INTRODUCTION

In the early 1950s, the need for a high-temperature hydraulic fluid evaluation circuit was dictated by the rigorous demands of predicted high performance aircraft hydraulic systems. It was intended that the new circuit function in concert with fluid bench tests and systems tests in evaluating new fluid lubricants. The new circuit was to fill the void between chemical bench tests, with their idealized environments, and simulated operational systems, with their expensive lubricity dependent hydraulic pumps.

By the end of 1956 the Air Force Materials Laboratory had developed a hydraulic fluid circuit capable of operating at temperatures to 700°F and pressures to 3000 psig (Reference 1). Hydraulic fluids were capable of being characterized for their thermal and shear stabilities and their corrosion, lacquering, and sludging tendencies. Also, the temperature limitations and expense of hydraulic pumps were eliminated in the first stage of circuit evaluations. To date, this circuit has been used to conduct thirty-three evaluations on fourteen hydraulic fluid candidates at temperatures ranging from 400 to 800°F.

One major system deficiency has become more significant with time, even though the circuit's performance has exceeded its design criteria. This deficiency is the fluid volume (approximately 2.5 gallons) required to conduct an evaluation. Experience has shown that it is extremely advantageous to conduct fluid circuit studies as early in the fluid development cycle as is practical. The fluid circuit results may then be utilized to guide a fluid development program's direction or to establish a continue or terminate decision on fluid development as early as possible. Consequently, a system capable of functioning with a relatively small research quantity of fluid would possess significant advantages in terms of money and time.

As a result of the limitations exhibited by the volume requirements of the original (homogenizer pump stand) fluid circuit, a program was started in 1963 to develop a small-volume, high-temperature fluid evaluation system. In 1966 the resulting circuit was ready for systems checkout tests and initial operation. The construction details and operational characteristics of the system were reported in 1967 (Reference 2).

Recent efforts, directed at refining the circuit to stabilize its operational characteristics, resulted in the investigation of the properties of several hydraulic fluid candidates and in establishing a correlation between the results produced by the SVHT stand and the homogenizer pump stand when operating under similar test profiles.

Two fluid candidates which had been evaluated in the homogenizer pump stand were selected for reevaluation in the SVHT stand. A deep dewaxed superrefined mineral oil, conforming to Military Specification MIL-H-27601, was selected as one fluid because of the number and temperature range (550 to 700°F) of experiments conducted with it. The second

fluid candidate, a structurally modified fluorosilicone, designated MLO-70-79, was selected for further study because of the property changes which resulted during an evaluation in the homogenizer pump stand at 600°F (Reference 6).

SECTION II

SYSTEM DESCRIPTIONS

The small-volume, high-temperature stand and its progenitor, the homogenizer pump stand, shall be described briefly by major mechanical components and modes of operation. Detailed descriptions of both pieces of equipment have been made previously (References 1 and 2).

1. The Homogenizer Pump Stand

The configuration of the homogenizer pump stand is presented schematically in Figure 1. The principal mechanical elements of the circuit are:

(1) Triplex pump, Manton Gaulin, Model 500-HP-KL6-3PA,
(2) Pneumatic Valve, Hammel-Dahl, with spline plug,
(3) Flowmeter, turbine type, with counter,
(4) Filter, 33 micron, 12 stainless steel discs of 5.53 square inches,
(5) Heat exchanger,
(6) Reservoir, remote type with rolling diaphragm,
(7) Corrosion specimen holder, and
(8) Lacquer indicator.

The circuit was constructed with stainless steel tubing. Appropriate circuit elements were housed in an environmental chamber. The system was completed with the addition of instrumentation to monitor and record desired functions and alarms to terminate circuit operation in the event preestablished system conditions are exceeded.

During the operation of the homogenizer pump stand, the fluid is pumped to the throttling valve which is adjusted to maintain a pump discharge pressure between 2900 and 3000 psig. Upon passing through the throttling valve, fluid pressure is reduced to approximately 25 psig. In continuing through the circuit, the fluid's rate of flow is measured, the fluid is filtered, and the fluid's temperature is adjusted prior to the fluid returning to the pump inlet. Temperature control is accomplished by sensing fluid temperature at the throttling valve outlet and employing the resulting thermocouple signal to regulate heat exchanger coolant flow.

The volume of the system circulating loop is approximately 1.5 gallons. The total system volume, with reservoir, is approximately 5.0 gallons (maximum). Pump discharge rates, which vary with fluid viscosity and discharge pressure, have been recorded to 5 gpm under steady state conditions. Fluid temperatures of 800°F have been achieved.

2. The Small Volume High Temperature Stand

The configuration of the small-volume high-temperature stand, hereafter referred to as the SVHT stand, is presented schematically in Figure 2. The principal circuit elements are:

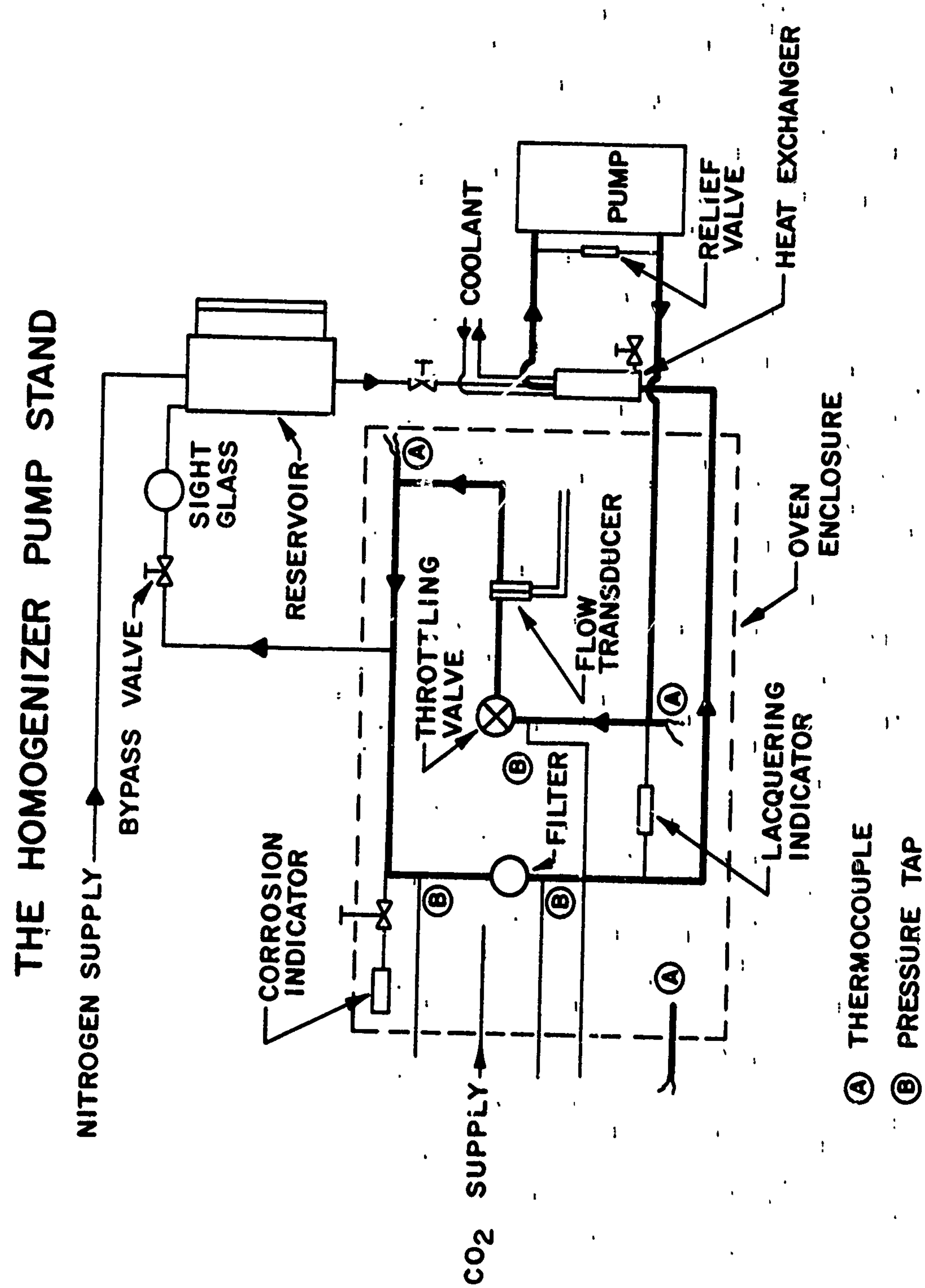
THE HOMOGENIZER PUMP STAND
NITROGEN SUPPLY
BYPASS VALVE
SIGHT GLASS
RESERVOIR
COOLANT
PUMP
RELIEF VALVE
HEAT EXCHANGER
OVEN ENCLOSURE
THROTTLING VALVE
FLOW TRANSDUCER
FILTER
LACQUERING INDICATOR
CORROSION INDICATOR
CO_2 SUPPLY
A
B
A THERMOCOUPLE
B PRESSURE TAP

FIGURE 1

THE SMALL VOLUME HIGH TEMPERATURE FLUID CIRCUIT

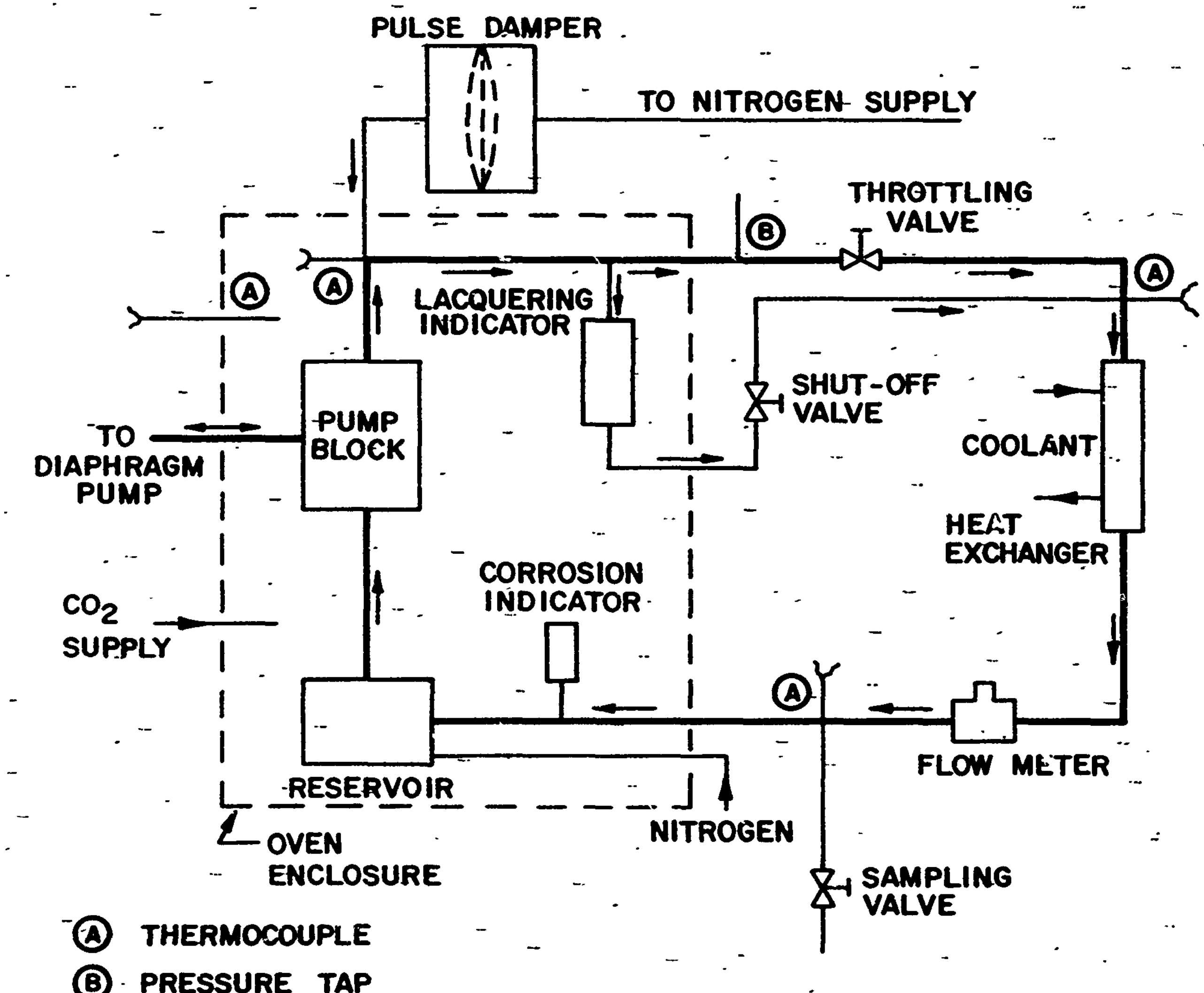

FIGURE 2

(1) Diaphragm pump, Lapp, Model CPS-45,
(2) Pump block, remote,
(3) Pulse damper,
(4) Throttling valve, Nupro, Model SS-4BMG,
(5) Flowmeter, Brooks, Model 4909-01,
(6) Reservoir, metallic bellows type,
(7) Lacquer indicator, and
(8) Corrosion specimen holder.

The circuit elements are appropriately connected with .375 diameter stainless steel tubing having a wall thickness of .065 inches. Where possible, all connections are made with either high pressure Aminco fittings or heliarc welds. A major portion of the circuit is constructed in a 3700 watt cylindrical oven whose inside dimensions are 23 inches deep and 24.5 inches in diameter. The addition of instrumentation and controls to monitor and record the desired parameters and to operate safety devices completes the system. An auxiliary piece of equipment, not shown on the schematic, is a high pressure gas booster which is used to produce a pressure of 3000 psig in the pulse damper.

During the operation of the SVHT stand, the discharge stroke of the diaphragm pump forces fluid to the center of the remote pump block. In the remote pump block, fluid pressure closes the bottom check valves and opens the top check valves to allow the fluid to flow toward the throttling valve. Prior to reaching the throttling valve the fluid is divided into two flow paths. Part of the fluid flows to the pulse damper and displaces the damper diaphragm against approximately 3000 psi nitrogen pressure. The remainder of the fluid continues to the throttling valve, which is adjusted to maintain a pump discharge pressure between 2900 and 3000 psig. From the throttling valve the fluid travels around the circuit through the flowmeter to the reservoir. At the reservoir the fluid is temporarily stored by displacing the stainless steel bellows. Further travel is impeded by the closed bottom check valves in the remote pump block.

During the intake stroke of the diaphragm pump, the top check valves of the remote pump block are closed by fluid pressure from the pulse damper. The high pressure nitrogen displaces the pulse damper diaphragm, forcing the fluid from the damper through the throttling valve. From the throttling valve the fluid passes through the flowmeter to the reservoir. The fluid stored in the reservoir during the pump discharge stroke is now expelled and joins the fluid from the flow meter to continue through the open check valves in the bottom of the remote pump block and back to the diaphragm pump.

Fluid temperature is sensed at the outlet of the throttling valve. This thermocouple signal is used to control fluid temperature by regulating the oven heaters. The fluid temperature is regulated to $\pm$ 5.0°F of the set temperature. Although the diaphragm pump operates at a low frequency, the flow and pressure modulating action produces an exceptionally even flow rate at a pressure which varies from 25 to 75 psi. The maximum flow rate of the system is approximately 0.5 gpm but operation is generally with a flow rate of 0.4 gpm or less.

3. Major System Differences

There are a number of features incorporated into the SVHT stand which make it superior to the homogenizer pump stand. Some of these features reflect the primary reasons for constructing the SVHT stand. Others represent solutions to problems experienced with the homogenizer stand.

As previously stated, one of the primary reasons for constructing the SVHT stand was to obtain the capability of evaluating small (one liter) quantities of experimental fluid lubricants earlier in their development cycle. The homogenizer pump stand has a circulating volume of approximately 1.5 gallons (5,678 ml) and a static reservoir volume in excess of 3.5 gallons (13,249 ml). The volume of the SVHT stand depends upon the use of one of two available reservoirs. The large reservoir, which is normally used, produces a circuit volume of 1550 to 1850 ml, depending upon the amount of bellows compression allowed. The small reservoir produces a circuit volume of less than 1000 ml.

Increased temperature capability was also a primary reason for constructing the SVHT stand. The homogenizer pump stand was intended to operate as high as 700°F. In practice the stand has operated at 800°F. The SVHT stand was designed to extend the test temperature capability to 1000°F. Although this temperature has not been reached in actual operation due to fluid limitations, high temperature checkout tests to 900°F have revealed no reasons for not reaching and exceeding the design temperature.

The reservoirs in each of the two circuits differ greatly in design and operation. The homogenizer pump stand reservoir uses a rolling diaphragm of elastomer-on-nylon mesh construction to separate the test fluid from the pressurant. The use of these materials precludes placing the reservoir in the oven environment or employing a flow-through type reservoir which would contain high temperature fluid. Consequently a remote or "dead leg" type reservoir was used. This reservoir supplies make-up fluid to the circulating portion of the system and must be mounted out of the high temperature areas. The major disadvantage with this reservoir configuration is the dilution of the fluid in the circulating system by lower temperature, unstressed fluid. This dilution occurs with any loss of fluid from the circulating system, whether it be from the collection of fluid samples or circuit leakage. The reservoir in the SVHT stand is an all metal, flow-through unit which uses a stainless steel bellows to separate the candidate fluid from the pressurant. This design allows the reservoir to be located inside the oven and does not result in dilution of the circulating fluid by low temperature, unstressed fluid since the total fluid charge is circulating at near maximum temperature.

The diaphragm pump in the SVHT stand is a major improvement over the triplex pump which is used in the homogenizer pump stand. Wear of the V-ring piston seals in the triplex pump constitutes a major source of fluid leakage, particularly during fluid experiments at 600°F and above. The use of the diaphragm pump eliminates all sliding seals in contact with the candidate fluid and, as a bonus, reduces the large fluid volume required by the triplex pump pressure block.

TABLE I

550°F FLUID STABILITY EXPERIMENT

FLUID: MIL-H-27601
CIRCUIT: SVHT Stand

Sample Test Hrs	Viscosity, (cs) 100°F	210°F	Flash Point, °F	Fire Point, °F	Neut. No MgKOH/gm
New	15.50	3.28	375	410	0.1
0					
2	14.77				0.1
5	14.51				
10	14.47				
23.4	14.41				
45	14.32				
75	14.22				0.1
100	14.56		375	410	0.1

CORROSION DATA

Material	Weight Change, Mg/cm^2	Specimen Surface
Aluminum, 2024	+0.07	Light Grey, dull
M-1 Tool Steel	+0.22	Deep Blue, reflective
4140 Steel	+0.25	Deep Blue, reflective
J02 Stainless	+0.03	No Change
440C Stainless	-0.03	Deep Blue, reflective
Titanium, RC70	-0.22	Light Tan, reflective

TABLE II

550°F FLUID STABILITY EXPERIMENT

FLUID: MIL-H-27601
CIRCUIT: Homogenizer Pump Stand

Sample Hr Pumped	Viscosity, (cs) 100°F	210°F	Flash Point, °F	Fire Point, °F	Neut. No MgKOH/gm
New	14.08	3.21	388	435	0.06
0	14.05	3.15	403	433	0.06
2	13.75	3.12	415	436	0.06
4	13.65	3.12	380	422	0.06
6	13.69	3.12	386	424	0.06
8	13.79	3.12	393	424	0.06
10	13.79	3.13	400	421	0.06
25a	13.69	3.12	398	423	0.06
25b	13.72	3.12	412	427	0.06
30	13.88	3.12	384	424	0.06
50	13.79	3.12	376	418	0.06
75	13.88	3.13	370	412	0.06
100	13.79	3.12	381	412	0.06

CORROSION DATA

Material	Weight Change, Mg/cm^2	Color	Texture
Aluminum 2024–T4	–0.27	None	None
M–1 Tool Steel	–0.07	Slightly Darkened	None
Chrome Moly Steel 4140	–0.03	Slightly Darkened	None
302 Stainless Steel	–0.05	None	None
440 Stainless Steel	–0.075	Slightly Darkened	None
Titanium RC130B	–0.055	Slightly Darkened	None

TABLE III

OPERATING DATA FOR A 100 HOUR, 550°F SVHT STAND FLUID STABILITY EXPERIMENT WITH MIL–H–27601 HYDRAULIC FLUID

		Maximum	Minimum	Average
Duration of Test	(HRS)	– –	– –	100
Pumping Rate	(GPM)	.3890	.3081	.3406
Reservoir Pressure	(PSIG)	8.0	0.0	0.25
Pump Discharge Pressure	(PSIG)			
Pulse HIGH		3120	2775	2943
Pulse LOW		3000	2675	2847
Temperatures	(°F)			
Before Throttling Valve		554	526	539
After Throttling Valve		568	440	552
Oven		632	564	569
Shear Cycles				6266
Fluid Composition at	(%)			
End of Test				
Original Charge				100
New Fluid Added				0

TABLE IV

OPERATING DATA FOR A 100 HOUR, 550°F
HOMOGENIZER STAND FLUID STABILITY EXPERIMENT
WITH MIL–H–27601 HYDRAULIC FLUID

		Maximum	Minimum	Average
Duration of Test	(HRS)	– –	– –	100
Pumping Rate	(GPM)	3.32	2.10	2.45
Resevoir Press	(PSIG)	NR	NR	NR
Pump Discharge Press	(PSIG)	NR	NR	NR
Filter Pressure Drop	(PSI)	153	0	68.4
Temperatures	(°F)			
Before Throttling Valve				NR
After Throttling Valve				550
Oven				NR
Shear Cycles				9700
Fluid Composition at	(%)			
End of Test				
Original Charge				27
New Fluid Added				73

NR = Not Reported

100°F VISCOSITY vs. TEST TIME
FOR
550°F EVALUATION OF
MIL-H-27601 FLUIDS

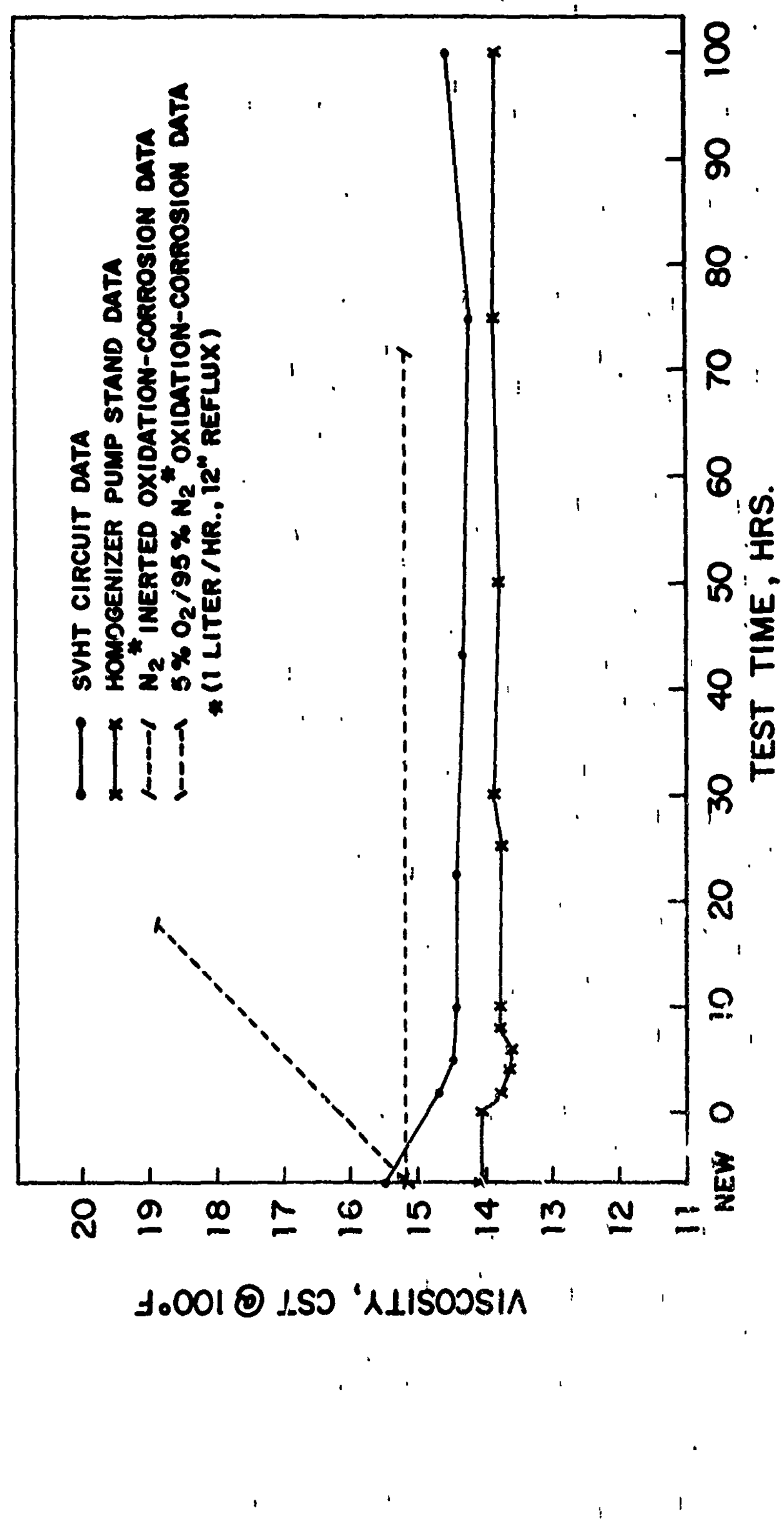

FIGURE 3

SECTION III

TEST RESULTS

A deep dewaxed superrefined mineral oil, conforming to Military Specification MIL-H-27601, and a perfluorosilicone, designated MLO-70-79, were evaluaced in the SVHT stand with time-temperature profiles analogous to the corresponding fluid experiments which were conducted in the homogenizer pump stand (References 3, 4, 5 and 6).

Evaluations of the mineral oil were made at time-temperature profiles of 100 hours at 550°F, successive 25 hour periods at 600 and 625°F, and 44.7 hours at 700°F.

Those parameters which exhibited significant deviations during the fluid evaluations in the two circuits were viscosity (100°F), flash point, fire point, and metal corrosion. In all of the tests, lacquer indicator piston forces were very small, indicating no significant tendency of the fluids to form lacquer films. Infrared spectrum analysis of fluid samples indicated that none of the test fluids had experienced gross molecular change.

1. The Evaluation of MIL-H-27601 at 550°F

The experimental data from the evaluation of MIL-H-27601 hydraulic fluid at 550°F for 100 hours in each of the test circuits are presented in Tables I and II. Operational data are presented in Tables III and IV.

Gross changes in viscosity were not experienced by the fluid in either of the test circuits. The fluid in the homogenizer pump stand exhibited a maximum loss in viscosity (100°F) of 3.06% at four hours of test. The SVHT stand fluid displayed an 8.26% viscosity (100°F) loss at 75 test hours, as shown in Figure 3.

The flash and fire points of the fluid samples from the two test stands are in good agreement, with no significant fluid degradation being indicated. The differences in the flash and fire points of the two sets of fluid samples are within the limits of precision of laboratory-to-laboratory reproducibility recognized by ASTM (Reference 7).

The corrosion specimen data from the two experiments exhibit severe differences. Each of the material specimens from the homogenizer pump stand lost weight. However, contradictory to these results, four of the six material specimens from the SVHT stand gained weight. Two of these specimens, M-1 tool steel and 4140 steel, experienced gains of 0.22 and 0.25 mg/cm^2, respectively. A weight change of 0.20 mg/cm^2 is considered an indicator of significant corrosive action.

2. The Evaluation of MIL-H-27601 at 600/625°F.

An evaluation of the candidate fluid in the homogenizer pump stand was

TABLE V

600/625°F FLUID STABILITY EXPERIMENT

FLUID: MIL–H–27601
CIRCUIT: SVHT Stand

Sample Test Hrs	Fluid Temp	Viscosity, (cs) 100°F	210°F	Flash Point, °F	Fire Point, °F	Neut. No. MgKOH/gm
New		15.05	3.3	390	435	0.1
0	600	15.03	3.3			0.1
5	600	14.99	3.3			
10	600	14.37	3.2			
15	600	14.53	3.2			0.1
25	600	14.64	3.2			
30	625	14.62	3.2			
35	625	14.76	3.2			
45	625	14.17	3.2			
50	625	13.08	3.0	365	420	0.1

CORROSION DATA

Materials	Weight Change, Mg/cm^2	Specimen Surface
Aluminum, 2024	+0.29	Dull Black
M–1 Tool Steel	+1.09	Deep Blue
4140 Steel	+0.83	Dull Black
302 Stainless	+0.68	Dull Areas
440C Stainless	+0.56	Dull Black
Titanium, RC70	–0.09	Deep Blue

TABLE VI

600/625°F FLUID STABILITY EXPERIMENT

FLUID: HIL-H-27601
CIRCUIT: Homogenizer Pump Stand

Sample Hr Pumped	Fluid Temp	Viscosity, (cs) 100°F	210°F	Flash Point, °F	Fire Point, °F
0	600	15.4	3.2	368	418
2	600	15.4	3.1	360	420
4	600	15.4	3.1	360	423
6	600	15.3	3.1	355	418
8	600	15.2	3.1	350	420
10	600	15.2	3.1	360	415
25	600	15.1	3.1	365	415
27	625	15.1	3.1	345	409
29	625	15.0	3.1	335	414
31	625	15.1	3.1	350	415
33	625	15.1	3.1	350	415
35	625	15.0	3.1	345	414
42.5	625	15.1	3.1	355	415

CORROSION DATA
(Taken After 19 and 42.5 Hr. Experiments)

Material	Weight Change, Mg/cm^2 Spec. #1	Spec. #2	Average	Color	Texture
Aluminum, 2024–T4	–1.304	–0.369	–0.837	None	None
M–1 Tool Steel	0	0	0	Slightly Darker	None
Chrome Moly Steel, 4140	–0.087	–0.413	–0.250	Slightly Darker	None
302 Stainless Steel	–0.348	–0.283	–0.316	Slightly Darker	None
440 Stainless Steel	–0.261	–0.261	–0.261	Slightly Darker	None
Titanium, RC–130B	–0.196	–0.196	–0.196	Slightly Darker	None

TABLE VII

OPERATING DATA FOR A 50 HOUR, 600/625°F

SVHT STAND FLUID STABILITY EXPERIMENT WITH

MIL–H–27601 HYDRAULIC FLUID

		Maximum	Minimum	Average
Duration of Test	(HRS)	– –	– –	(25/25) 50
Pumping Rate	(GPM)	.3797	.2989	.3458
Reservoir Pressure	(PSIG)	35	0	14.2
Pump Discharge Pressure	(PSIG)			
Pulse MAX		3120	2910	3011
Pulse MIN		3025	2780	2939
Temperatures	(°F)*			
Before Throttling Valve		605/616	576/581	586/604
After Throttling Valve		622/642	590/600	600/626
Oven		689/667	612/642	620/648
Shear Cycles			(1449/1421)	2870
Fluid Composition at	(%)			
End of Test				
Original Charge				100
New Fluid Added				0

*600°F Data/625°F Data

TABLE VIII

N_2 INERTED OXIDATION CORROSION TEST

FLUID: MIL–H–27601

25 Hours at 600°F
25 Hours at 625°F

CORROSION DATA*

Material	Weight Change, Mg/cm^2, Avg.	Appearance
M–1 Tool Steel	+0.03	Dark Brown Spots
440C Stainless Steel	+0.01	Dark Blue
Aluminum	+0.02	No Change
Titanium	+0.01	Light Tan
302 Stainless Steel	0.00	Light Tan/Blue
4140 Steel	+0.02	Yellow Blue

FLUID DATA*

	New	After Test
Viscosity (100°F), (cs)	15.5	14.2
Flash Point, °F	390	365
Fire Point, °F	420	405
Average Fluid Loss 4.6%		

*Average of 2 Tests

100° F VISCOSITY vs. TEST TIME
FOR
600/625° F EVALUATION OF
MIL-H-27601 HYDRAULIC FLUIDS

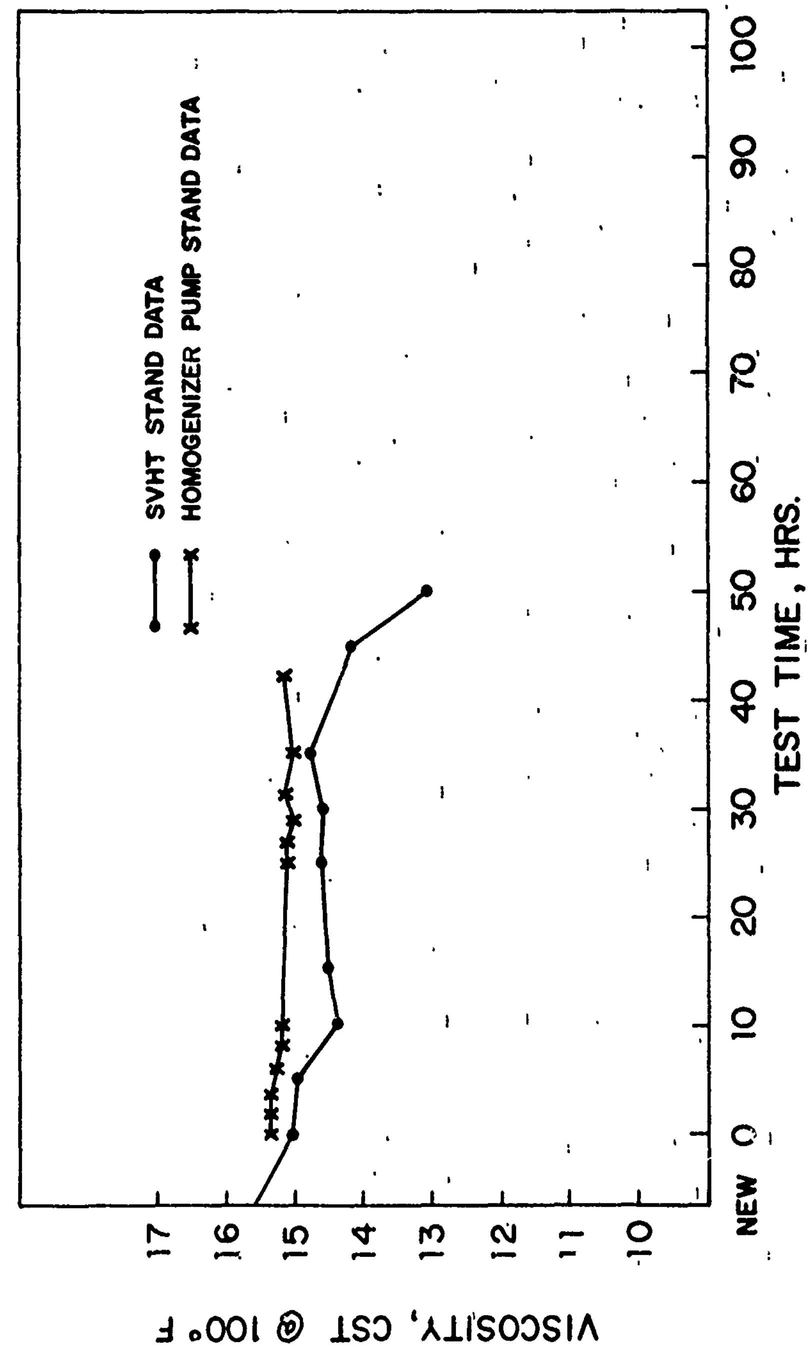

FIGURE 4

conducted for 25 hours at 600°F followed by 17.5 hours at 625°F. The results of this study were inconclusive since it is suspected that dilution of the circulating fluid may have masked fluid property changes in the 625°F phase of the test (Reference 4).

An evaluation of the candidate fluid was made in the SVHT stand for 25 hours at 600°F followed by 25 hours at 625°F. Fluid sample properties and corrosion data are presented in Tables V and VI. Operational data are shown in Table VII.

Fluid degradation in the homogenizer pump stand was suspected from a decrease in sample flash point, but the history of this property is not easily followed in the SVHT stand due to the allowable size of fluid samples. Viscosity (100°F) is determined for each fluid sample, and, from Figure 4, there is evidence of incipient fluid degradation during the 625°F phase. Until the test had progressed for 35 hours there were no gross changes in viscosity. However, the 45 and 50 hour fluid samples indicate that, after 35 test hours, a decrease in viscosity started, which resulted in a 10.4% loss during the phase at 625°F and at 13.1% loss for the test. It should be noted from Figure 5 that during the tests, the SVHT stand fluid was not diluted while the homogenizer pump stand fluid was diluted 76%.

Even though the fluid in the homogenizer pump stand was grossly diluted during test, a comparison of corrosion specimen data from the two systems is of consequence. The weight changes of the corrosion specimens from the two tests differed greatly. Specimens from the homogenizer pump stand exhibited losses in weight, whereas the specimens from the SVHT stand, in general, gained weight. These weight changes in the materials specimens from the two test stands were, in the main, significant changes - 0.20 mg/cm^2 and greater. To establish a baseline for these diverse results, duplicate tests were conducted with an oxidation-corrosion apparatus and a materials profile as close to the materials in the fluid test stands as was obtainable. Dry, laboratory grade, nitrogen was bubbled through the candidate fluid during test, instead of air or an oxygen containing gas mixture. The duplicate tests were conducted for 25 hours at 600°F followed by 25 hours at 625°F. The test results, shown in Table VIII reveal that none of the material specimens lost weight. The increases in specimen weight which were exhibited were small, with none approaching the significant level of 0.20 mg/cm^2. These small corrosion specimen weight changes are attributed to the superior inert atmosphere provided by the high purity nitrogen of the oxidation-corrosion apparatus - an operational environment which is very difficult to achieve in a mechanical system of complex geometry.

3. <u>The Evaluation of MIL-H-27601 at 700°F</u>

The candidate hydraulic fluid was evaluated in the SVHT stand at 700°F for 44.1 hours to provide a comparison with the 27 hour, 700°F test of the fluid in the homogenizer pump stand. Operational pressure instabilities which occurred during the SVHT stand test were found to be caused by excessive fluid volume. In alleviating the problem, too much fluid was removed from the circuit. Consequently, 70 ml of fluid was returned to the system. This resulted in the fluid composition being only 88.8 percent original after 29 test hours. The result of this dilution would only be evident in the last (44.1 hour) fluid sample, as is shown in Figure 5.

FIGURE 5

TEST CIRCUIT FLUID DILUTION HISTORIES MIL-H-27601 & MLO-70-79

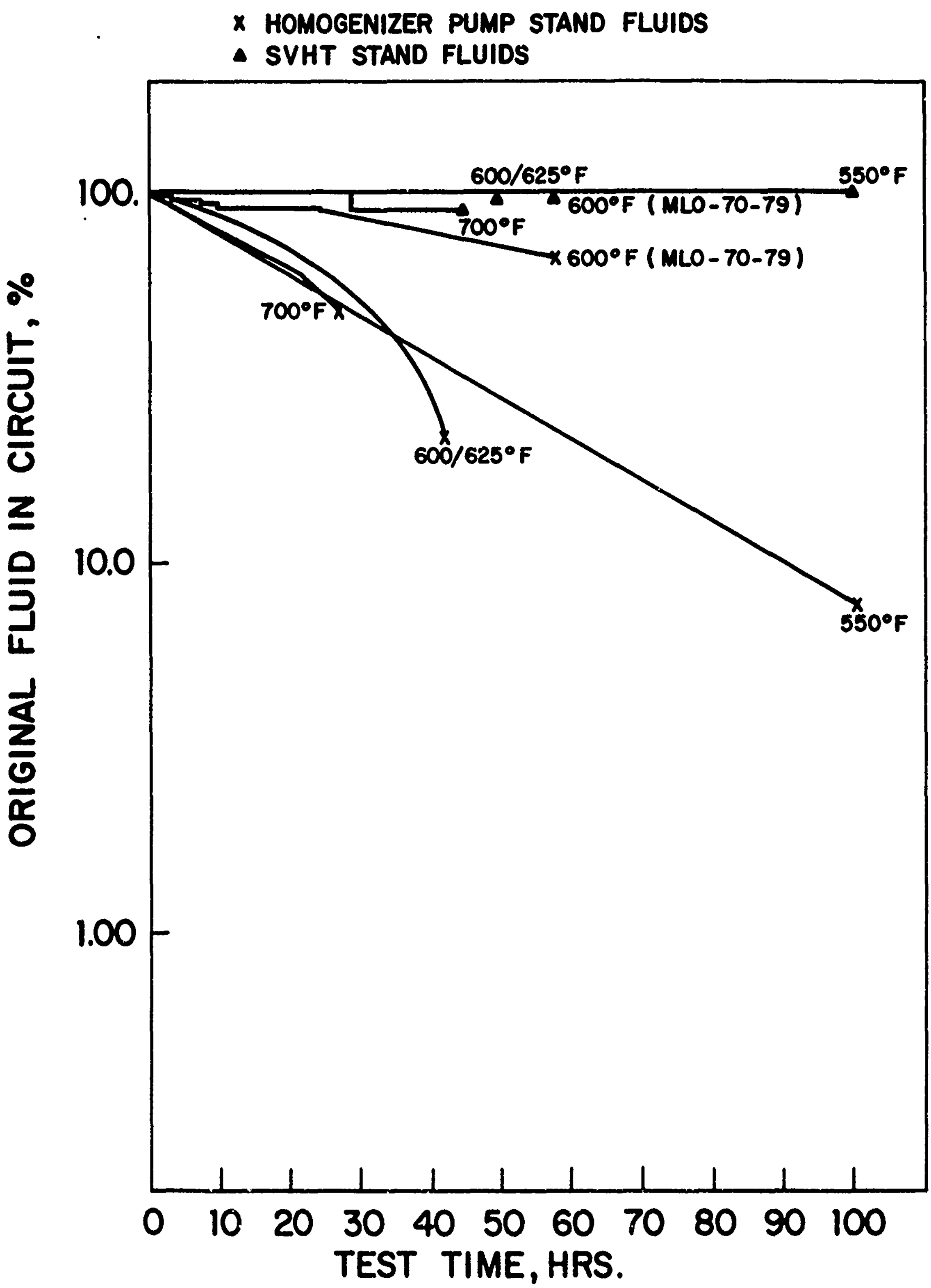

TABLE IX

700°F FLUID STABILITY EXPERIMENT

FLUID: MIL–H–27601
CIRCUIT: SVHT Stand

Sample Test Hrs	Viscosity, (cs) 100°F	210°F	Flash Point, °F	Fire Point, °F	Neut. No. MgKOH/gm
New	15.50	3.28	390	420	
0	14.47	3.20			0.1
2	13.08	3.03			
5	14.31	3.10			0.1
11	12.46	2.88			0.1
25	10.90	2.80			
44.1	11.20	2.79	260	340	0.1

CORROSION DATA

Material	Weight Change, Mg/cm^2	Specimen Surface
Aluminum, 2024	+0.01	Light Yellow, reflective
M–1 Tool Steel	+0.26	Black, dull
4140 Steel	+0.40	Black, dull
320 Stainless	– 0.02	Yellow/Blue, reflective
440C Stainless	+0.30	Grey, dull
Titanium RC70	–0.09	Black, reflective

TABLE X

700°F FLUID STABILITY EXPERIMENT

FLUID: MIL–H–27601
CIRCUIT: Homogenizer Pump Stand

Sample Hr Pumped	Viscosity, (cs) 100°F	210°F	Flash Point, °F	Fire Point, °F	Neut. No. MgKOH/gm
New	13.79	3.29	376	428	0.07
0	13.19	3.15	340	403	0.07
2	12.50	2.97	290	391	0.07
4	11.81	2.89	255	377	0.07
6	11.45	2.86	215	367	0.07
8	11.32	2.86	170	357	0.07
10	11.15	2.85	175	350	0.07
25	10.69	2.74	195	332	0.07
27	10.69	2.73	162	308	0.07

CORROSION DATA

Material	Weight Change, Mg/cm^2	Color	Texture
Aluminum 2024–T4	0	None	None
M–1 Tool Steel	0	Much Darker	None
Chrome Moly Steel 4140	0	Very Much Darker	None
302 Stainless Steel	–0.088	Slightly Darker	None
440 Stainless Steel	–0.043	Slightly Darker	None
Titanium RC130B	0	Much Darker	None

TABLE XI

OPERATING DATA FOR A 44.1 HOUR, 700°F

SVHT STAND FLUID STABILITY EXPERIMENT

WITH MIL-H-27601 HYDRAULIC FLUID

		Maximum	Minimum	Average
Duration of Test	(HRS)	– –	– –	44.1
Pumping Rate	(GPM)	.3787	.0605	.2159
Reservoir Pressure	(PSIG)	30	5	21
Pump Discharge Pressure	(PSIG)			
Pulse Max		3000	2800	2893
Pulse MIN		2900	2725	2829
Temperatures	(°F)			
Before Throttling Valve		700	650	666
After Throttling Valve		730	670	698
Oven		785	712	735
Shear Cycles				2419
Fluid Composition at	(%)			
End of Test				
Original Charge				88.8
New Fluid Added				11.2

TABLE XII

OPERATING DATA FOR A 27 HOUR, 700°F
HOMOGENIZER STAND FLUID STABILITY EXPERIMENT
WITH MIL-H-27601 HYDRAULIC FLUID

		Maximum	Minimum	Average
Duration of Test	(HRS)	--	--	27
Pumping Rate	(GPM)	4.7	3.2	3.7
Reservoir Pressure	(PSIG)			80
Pump Discharge Pressure	(PSIG)			3000
Filter Pressure Drop	(PSI)	28.5	14.0	19.7
Temperatures	(°F)			
Before Throttling Valve				NR
After Throttling Valve				700
Oven				660
Shear Cycle				3960
Fluid Composition at	(%)			
End of Test				
Original Charge				51
New Fluid Added				49

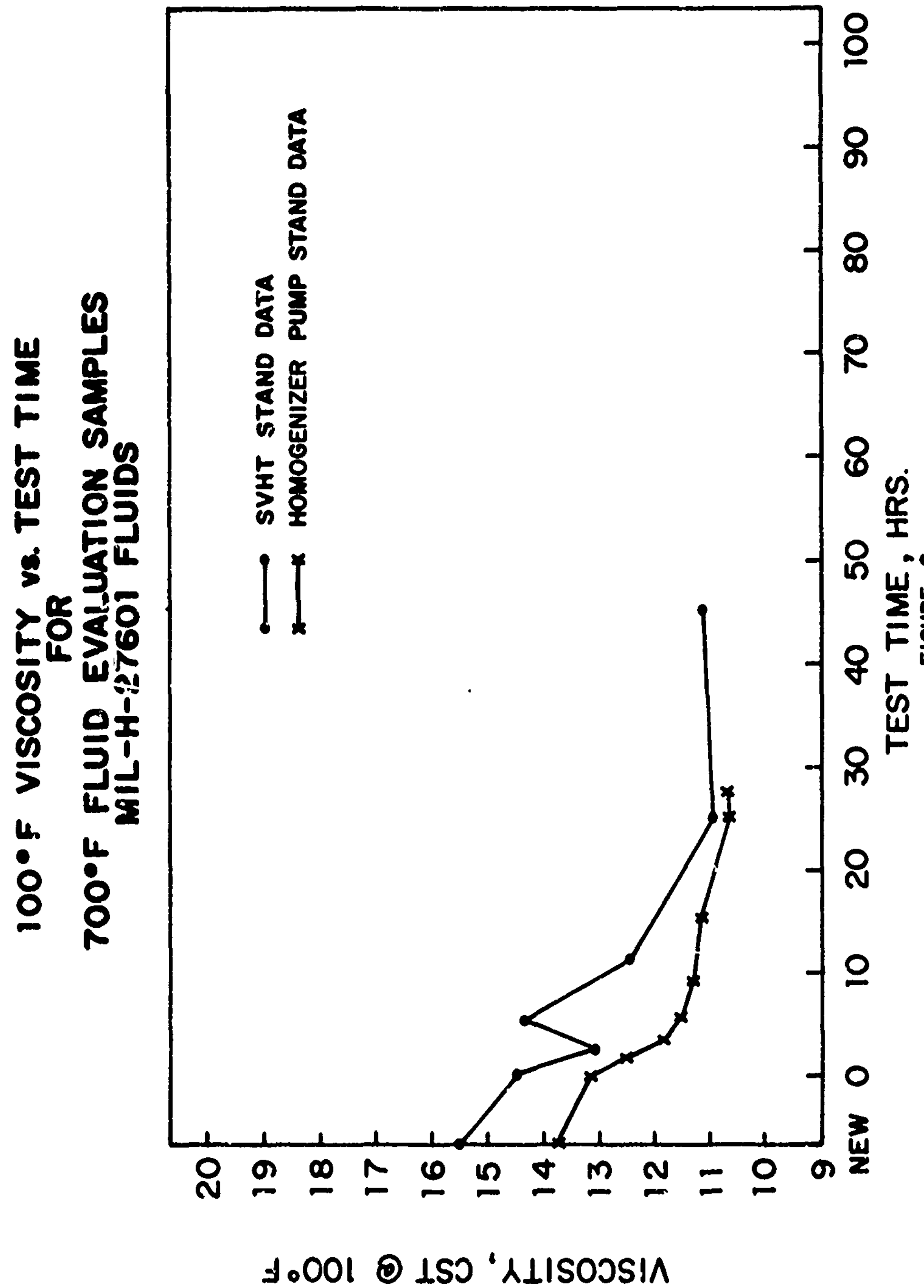
100°F VISCOSITY vs. TEST TIME
FOR
700°F FLUID EVALUATION SAMPLES
MIL-H-27601 FLUIDS
SVHT STAND DATA
HOMOGENIZER PUMP STAND DATA
VISCOSITY, CST @ 100°F
20
19
18
17
16
15
14
13
12
11
10
9
NEW
0
10
20
30
40
50
60
70
80
90
100
TEST TIME, HRS.

FIGURE 6

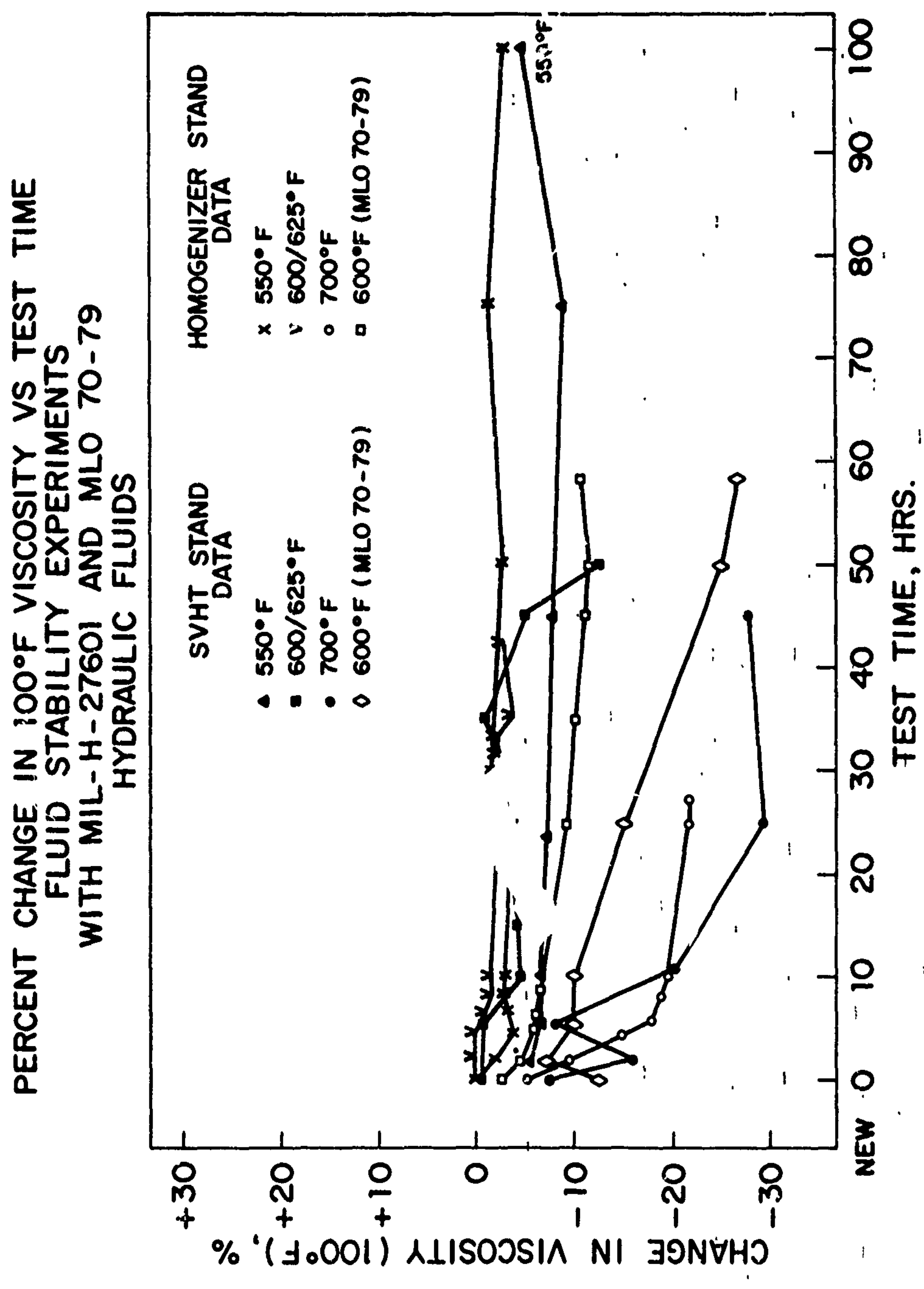
PERCENT CHANGE IN 100°F VISCOSITY VS TEST TIME
FLUID STABILITY EXPERIMENTS
WITH MIL-H-27601 AND MLO 70-79
HYDRAULIC FLUIDS
SVHT STAND DATA
550°F
600/625°F
700°F
600°F (MLO 70-79)
HOMOGENIZER STAND DATA
550°F
600/625°F
700°F
600°F (MLO 70-79)
CHANGE IN VISCOSITY (100°F), %
+30
+20
+10
0
-10
-20
-30
NEW
0
10
20
30
40
50
60
70
80
90
100
TEST TIME, HRS.

FIGURE 7

TABLE XIII

N_2 INERTED OXIDATION CORROSION TEST

FLUID: MIL-H-27601

50 Hours at 700°F

CORROSION DATA*

Material	Weight Change, Mg/cm^2, Avg.	Specimen Surface
M-1 Tool Steel	+0.09	Very Light Grey
440C Stainless Steel	+0.09	Medium Grey
Aluminum	+0.07	Light Tan Edges
Titanium	+0.03	Purple/Blue
302 Stainless Steel	+0.02	Light Blue
4140 Steel	+0.09	Medium Grey

FLUID DATA*

	New	After Test
Viscosity (100°F), (cs)	15.5	12.1
Flash Point, °F	390	310
Fire Point, °F	420	380

Average Fluid Loss 7.1%

*Average of 2 Tests

TABLE XIV

600°F FLUID STABILITY EXPERIMENT

FLUID: MLO-70-79, Lot #5
CIRCUIT: SVHT Stand

Sample Test Hrs	Viscosity, (cs) 100°F	210°F	Flash Point, °F	Fire Point, °F	Neut. No. MgKOH/gm
New	28.6	6.32	445	525	0.1
0	25.0				
2	26.6				
5	26.0				
10	25.7				
25	24.1				
50	21.6				
58	20.7		390	440	

CORROSION DATA

Material	Weight Change, Mg/cm^2	Specimen Surface
Aluminum, 2024	+0.22	No Change
M-1 Tool Steel	+0.80	Dark Grey, reflective
4140 Steel	+0.17	Purple/Green, reflective
302 Stainless	+0.08	No Change
440C Stainless	+0.35	Medium Grey, reflective
Titanium RC70	+0.05	Slight Tan, reflective

TABLE XV

600°F FLUID STABILITY EXPERIMENT

FLUID: MLO-70-79, Lot #4
CIRCUIT: Homogenizer Pump Stand

Hours Pumped	Viscosity (cs) 100°F	210°F	450°F	Flash Point, °F	Fire Point, °F	Neut. No. MgKOH/gm	Insolubles
New	26.85	5.87	1.36	450	518	0.01	--
0	26.05	5.73	1.33	445	518	0.05	--
2	25.61	5.70	1.32	445	515	0.06	--
4	25.18	5.61	1.31	430	510	0.08	--
6	25.17	5.61	1.32	430	505	0.08	--
8	24.99	5.61	1.31	425	500	0.08	--
10	25.03	5.56	1.32	419	495	0.09	--
25	24.32	5.50	1.31	390	485	0.13	--
35	24.23	5.41	1.31	395	490	0.18	Measurable
45	23.82	5.45	--	428	480	0.20	Measurable
50	23.62	5.42	--	400	482	0.21	Measurable
58	23.97	5.45	--	375	480	0.21	Measurable

CORROSION DATA

Material	Weight Change, Mg/cm^2 Spec. #1	Spec. #2	Avg.	Color	Texture
Aluminum, 2024-T4	-0.647	-0.167	-0.407	Slightly Darkened	No Change
M-1 Tool Steel	+0.114	+0.023	+0.069	Dark - purple tint	No Change
Chrome Moly Steel	+0.067	0.0	+0.034	Dark - purple tint	No Change
302 Stainless Steel	0.0	0.0	0.0	Slightly Darkened	No Change
440 Stainless Steel	+0.043	0.0	+0.022	Dark - purple tint	No Change
Titanium, RC130B	0.0	-0.066	-0.033	Slightly Darkened	No Change

TABLE XVI

OPERATING DATA FOR A 58 HOUR, 600°F
SVHT STAND FLUID STABILITY EXPERIMENT
WITH MLO-70-79 (FLUORINATED SILICONE)

		Maximum	Minimum	Average*
Duration of Test	(HR)	--	--	58
Pumping Rate	(GPM)	.3571	.1923	.2479
Reservoir Press	(PSIG)	20	0	10.6
Pump Discharge Press	(PSIG)			
Pulse Max		3125	2800	925
Pulse Min		3050	2725	840
Temperatures	(°F)			
Before Throttling Valve		587	580	583
After Throttling Valve		610	590	602
Oven		633	616	625
Shear Cycles		--	--	2212
Fluid Composition at	(%)			
End of Test				
Original Charge				100
New Fluid Added				0

*Average of 43 Data Recordings

TABLE XVII

OPERATING DATA FOR A 58 HOUR, 600°F
HOMOGENIZER PUMP STAND FLUID STABILITY EXPERIMENT
WITH MLO-70-79 (FLUORINATED SILICONE)

		Maximum	Minimum	Average*
Duration of Test	(HR)	– –	– –	58
Pumping Rate	(GPM)	3.53	0.57	2.55
Reservoir Pressure	(PSIG)	65	60	61.2
Pump Discharge Pressure	(PSIG)	3000	2825	2979
Pressure Before Filter	(PSIG)	360	70	203.8
Pressure After Filter	(PSIG)	60	45	53.4
Filter Pressure Drop	(PSI)	305	12	150.2
Pressure Between Piston Seals	(PSIG)	250	200	229
Temperatures	(°F)			
Before Throttling Valve		593	556	583
After Throttling Valve		605	575	596
Oven		687	590	626
Shear Cycles		– –	– –	5916
Fluid Composition at	(%)			
End of Test				
Original Charge		– –	– –	60.7
New Fluid Added		– –	– –	39.3

*Average of 40 Readings taken at least 1 Hr. apart

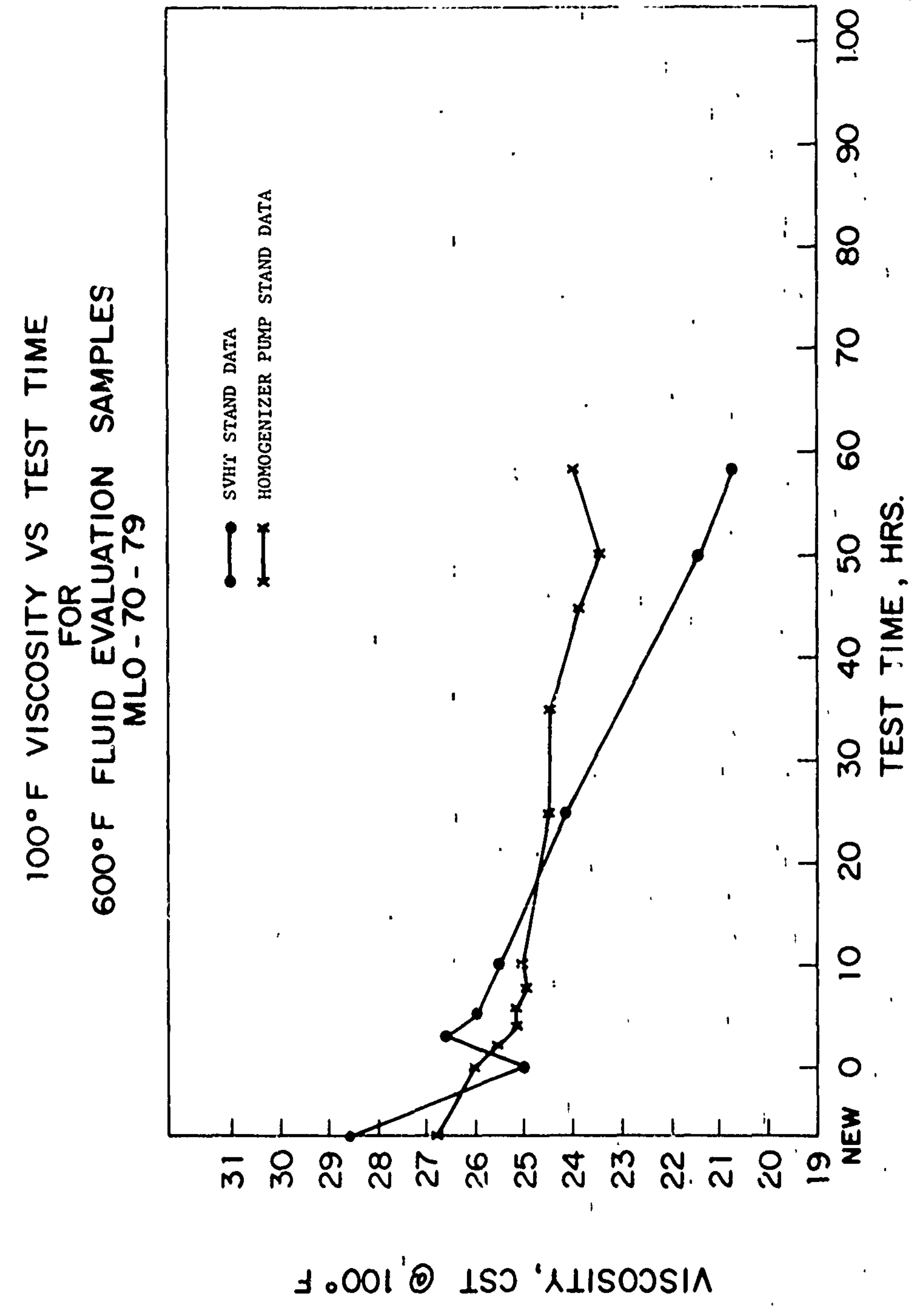
100°F VISCOSITY VS TEST TIME
FOR
600°F FLUID EVALUATION SAMPLES
MLO-70-79
SVHT STAND DATA
HOMOGENIZER PUMP STAND DATA
VISCOSITY, CST @ 100° F
31
30
29
28
27
26
25
24
23
22
21
20
19
NEW
0
10
20
30
40
50
60
70
80
90
100
TEST TIME, HRS.

Figure 8

The data from the evaluations of the candidate fluid in the two test circuits are presented in Tables IX and X. Operating data for the two tests are shown in Tables XI and XII.

The results of sample viscosity (100°F) determinations from the two circuits are shown in Figure 6. As in previous viscosity determinations, greater changes occur in the SVHT stand, as shown in Figure 7, with the actual changes in the homogenizer stand suspected of being masked by dilution (Figure 5).

The flash points of the fluid samples from the two evaluation circuits differ considerably in the 700°F tests. The flash point of the SVHT stand fluid decreases 33.4 percent in 44.1 test hours, with 2419 shear cycles. The flash point of the homogenizer pump stand fluid decreases 57 percent in 27 test hours, with 3960 shear cycles.

The corrosion specimen weight changes resulting from the homogenizer pump stand test were unlike the specimen changes from the previous examinations at 550 and 600/625°F. Four of the six material specimens showed no weight changes and the remaining two materials - 302 stainless steel and 440 stainless steel - exhibited low weight changes but did appear to have experienced significant corrosive attack. These results were in close agreement with the specimen weight changes obtained from the nitrogen-inerted, oxidation-corrosion test results at 700°F shown in Table XIII.

A comparison of the temperatures in the operational data, Tables XI and XII, reveals an unusual condition existed during the homogenizer pump stand test. The maximum fluid temperature was 40°F greater than the oven temperature. The SVHT stand operational data shows the average maximum fluid temperature was exceeded 37°F by the average oven temperature. Similar differences are noted for all of the previous fluid experiments. No explanation has been found for this temperature shift during the homogenizer pump stand test.

4. The Evaluation of a Fluorosilicone at 600°F

A structurally modified fluorosilicone, designated MLO-70-79, was selected as a candidate for comparing the results produced by the two fluid evaluation circuits with a non-petroleum fluid. The candidate fluid was subjected to a 600°F operational environment in each of the two circuits for a period of 58 hours. The results of property determinations and corrosion data on the fluid samples from the SVHT stand and homogenizer pump stand are presented in Tables XIV and XV, respectively. Operating data are presented in Tables XVI and XVII.

The viscosities (100°F) of fluid samples are presented for comparison in Figure 8. Relative changes in the viscosities (100°F) of fluid samples are presented in Figure 7. The fluid in the SVHT stand exhibited a 26.5% decrease in viscosity after 58 hours of test. The fluid in the homogenizer

PERCENT CHANGE IN FLASH POINT VS TEST TIME
FLUID STABILITY EXPERIMENTS
WITH MIL-H-27601 AND MLO 70 - 79
HYDRAULIC FLUIDS

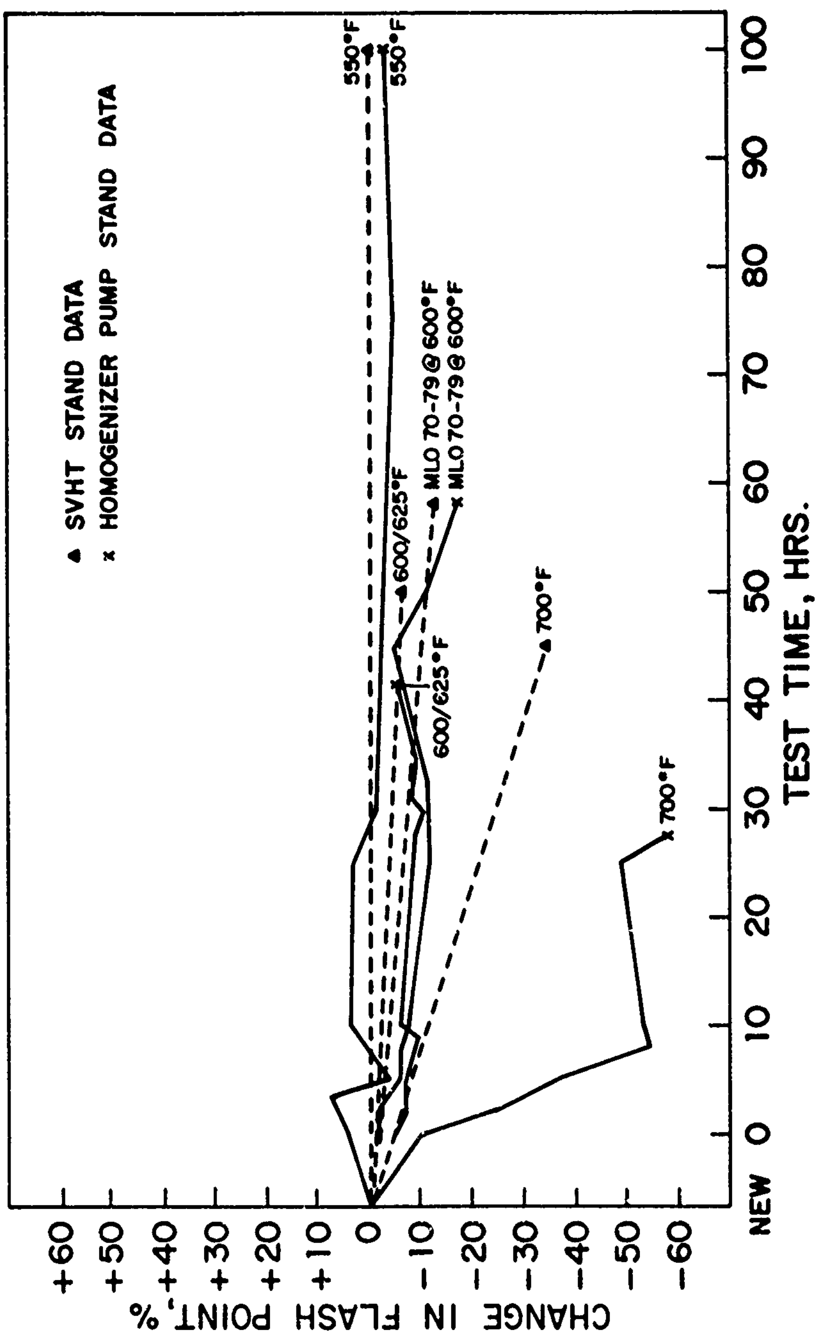

FIGURE 9

pump stand experienced a 10.7% decrease in viscosity in the same period of time.

The general pattern of fluid sample flash points, established between the two circuits during the evaluations of the petroleum fluid is present in the results of the fluorosilicone fluid tests. A comparison of the end-of-test (58 hour) fluid sample flash points in Figure 9, shows a 12.4 percent (55°F) decrease in the SVHT stand fluid and a 16.7 percent (75°F) decrease in the homogenizer pump stand fluid.

The corrosion specimen data from the test of MLO-70-79 in the SVHT stand is similar to the petroleum fluid data in that all material specimens gained weight. The data from the homogenizer pump stand were more varied. Three of the six specimens - M-1 tool steel, 4140 steel, and 440 stainless steel - gained averages of 0.069, 0.034, and 0.022 mg/cm^2, respectively. Two material samples - 2024 aluminum and titanium RC130B - lost relative weight of 0.0407 and 0.033 mg/cm^2, while the 302 stainless steel specimen remained unchanged. The reported appearance of the corrosion specimens from the two circuits was quite similar.

SECTION IV

DISCUSSION OF RESULTS

The deep dewaxed superrefined mineral oil (MIL-L-27601) exhibited no significant response to the combined stresses of the thermal/shear environment until experiencing a temperature of 625°F. Theretofore, all fluid property changes should be considered normal and insignificant. However, during the fluid's 25-hour residence at 625°F, a distinct decaying trend was established in the viscosity (100°F) history, as shown in Figure 4. This loss of viscosity is considered incipient fluid degeneration. This judgement is supported by the results of the following investigation at 700°F. At this higher temperature, the loss of viscosity (100°F) is immediate and decisive, with a 29 percent decrease occurring in 25 hours. Although not responsive at 625°F, the fluid flash point decreases 34 percent during the 700°F test (Figure 9). This characterization of the candidate fluid clearly indicates that is maximum operational temperature in an inerted system is approximately 600°F.

The structurally modified fluorosilicone fluid, MLO-70-79, was characterized only at 600°F, primarily for comparative purposes. The significant decreases in both viscosity and flash point are enough indication that the operating temperature of this fluid is below 600°F in an inerted system.

Major differences in results produced by the two fluid circuits are suggested from the comparative data. By limiting cases to a given fluid being evaluated in each of the test circuits at a specific time-temperature profile, these apparent differences may be summarized as follows:

1. The homogenizer pump stand produces a more severe environment for the fluid than does the SVHT stand.

2. The homogenizer pump stand produces greater changes in fluid flash point than does the SVHT stand.

3. The SVHT stand produces greater apparent changes in fluid viscosity than does the homogenizer pump stand.

4. Corrosion specimen results from the two circuits exhibit great divergence with the specimens from the homogenizer pump stand experiencing weight losses and the specimens from the SVHT stand experiencing weight increases.

From the operating temperatures, it is concluded that the thermal environments produced by the two circuits are comparable. Also, since proof is beyond the scope of this program, it is assumed that the shear experienced by the fluid in each of the circuits is equal for equal pressure differentials across the throttling valves. It then follows, that for a specific time-temperature profile, the homogenizer pump stand produces a more severe fluid environment since its fluid shear cycle rate is greater.

The differences in fluid flash points from the two circuits are also

attributable to the differences in shear cycle rates. Thermal environments have been found comparable. A further comparison of the data reveals that the homogenizer pump stand subjects its fluid to between 1-1/2 and 2-1/2 times as many shear cycles as does the SVHT stand for a given time period. If fluid shear cycles are accepted as proportional to test time, the candidate fluid in the homogenizer pump stand at 700°F would have experienced the same number of shear cycles in 16.5 hours as the fluid in the SVHT stand did in 44.1 hours. From Figure 7, the change in flash point experienced by the fluid in the SVHT stand in 44.1 hours is shown to be duplicated by the fluid in the homogenizer pump stand in approximately 4.5 hours. In the results of the 700°F homogenizer pump stand test, it is reported that a pressure instability produced a 9.75 hour prerun adjustment period, with 5.25 hours of this at full test conditions. With the fluid test data altered to include this substantial prerun period the picture would change drastically. The 2419 shear cycles would be produced in the same length of time; however, the time of similar fluid flash point change is now 14.25 hours instead of the 4.5 hours. A time of 14.25 hours in the homogenizer pumpt stand is very agreeable with the theoretical 16.5 hours required to match shear cycles. If the same logic is applied to the results from the evaluations of the fluorosilicone fluid at 600°F, 22.6 hours in the homogenizer pump stand are required to equal the number of shear cycles accumulated by the fluid in the SVHT stand in 58 hours. Figure 7 shows that the total change in SVHT stand fluid flash point - 12.5 percent, is reached by the fluid in the homogenizer pump stand in 23 hours. This is excellent agreement with the theoretical time of 22.6 hours based solely on the number of shear cycles. However, it is not suggested that the differences in fluid flash points are totally the result of shear cycle rates.

It has been concluded that the homogenizer pump stand produces a more severe environment than the SVHT stand within the context of a given test profile. It then follows that this more severe environment would manifest itself with greater changes in viscosity occurring in the homogenizer pump stand fluids. However, in every set of comparative tests, the data reveals that the greatest viscosity (100°F) changes occurred in the SVHT stand. The reasons for these apparent gross discrepancies may be deduced from Figure 5. Except for one case of operator error, the fluid in the SVHT stand was not diluted during test. In contrast, the circulating fluid in the homogenizer pump stand was diluted between 34 and 92 percent in each test. The results of the evaluation of the fluorosilicone fluid, MLO-70-79, show that samples of undiluted fluid from the SVHT stand exhibited a nearly linear decrease in viscosity (100°F) throughout the experiment. The fluid samples from the homogenizer pump stand displayed a significant decrease in viscosity (100°F) in the early test hours (small dilution), but, as the experiment progressed and dilution of the circulating fluid increased (Figure 5), the change in viscosity (100°F) became much less than the change in the SVHT stand fluid. At the end of each experiment, change in viscosity (100°F) of the SVHT stand fluid was approximately 14.8 percent greater than that of the homogenizer pump stand fluid. Also, the SVHT stand fluid was undiluted while the homogenizer pump stand fluid was diluted approximately 34 percent (Figure 5). The wide deviation produced by the two test stands in fluid viscosity results is therefore primarily attributed to the severe dilution of the circulating fluid in the homogenizer pump stand.

It should be noted that fluid flash point and fluid viscosity may

react quite differently to dilution. The dilution of one fluid by another, having a measurably different viscosity usually results in an immediate normalizing change in the viscosity of the mixture, assuming no chemical reaction. However, a low flash point fluid may be diluted several orders of magnitude by a high flash point fluid with the resulting mixture exhibiting essentially the same flash point as the low flash point constituent.

The difference in corrosion specimen results produced by the two circuits has been discussed. These results are believed to be due more to handling techniques than to circuit characteristics. The corrosion specimens used in the homogenizer pump stand were lightly burnished at the end of each test to remove surface accumulations. The corrosion specimens from the SVHT stand received no burnishing but were rinsed in solvent and vacuum dried for 50 hours. A further study of corrosion specimen results is proposed.

CONCLUSIONS

This investigation of a deep dewaxed superrefined mineral oil and a modified fluorsilicone in a new small volume high temperature (SVHT) stand has shown the following:

1. The mineral oil candidate (MIL-H-27601) exhbits no significant degradation after experiencing the combined stresses of the shear/thermal environment at 600°F for 25 hours.

2. The fluorsilicone fluid (MLO-70-79) exhibited a 27.5 percent viscosity loss after 58 hours at 600°F.

3. The SVHT stand is superior to its predecessor and represents a meaningful advance in fluid lubricant test devices.

4. The differences in corrosion results obtained from the two test circuits could not be correlated or resolved; however, it is believed they are attributable to handling techniques.

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